

Recommended Specifications for Microchemical Apparatus

Carius Method

Committee for the Standardization of Microchemical Apparatus, Division of Analytical Chemistry,
American Chemical Society

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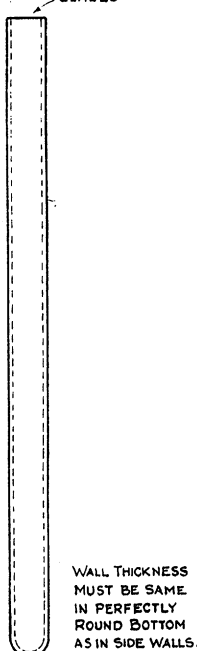


Figure 1. Combustion Tube

RECOMMENDED specifications for microchemical apparatus used in carbon-hydrogen, Dumas nitrogen, halogen, and sulfur determinations (3) and in the micro-Kjeldahl nitrogen determination (5), for the all-metal needle valve of the Hershberg-Southworth type (4), and for a new design of rubber stoppers for microchemistry (2), as well as the proposed program for the future (1), have been published. This report includes recommendations for the combustion furnace and two types of combustion tubes for the micro-Carius procedure (halogen, sulfur, arsenic).

Combustion Furnace. A portable furnace suitable for conducting the reactions in the Carius procedure should have at least four wells of approximately

more than $\pm 5^\circ$ C. from the operating temperature. [The temperature of an electrically heated furnace must be maintained at 310° C. with 100 volts (minimum).] The furnace temperature should be adjustable. A device that shows when the furnace is in operation and a temperature indicator should be provided.

The furnace must be equipped with safety devices to confine broken glass in the event of an explosion.

Combustion Tubes (Bomb Tubes). Recommended specifications for the combustion tubes take into account the fact that the details of the procedure vary, particularly in the amount of fuming nitric acid used. Accordingly, two types of tubes are recommended—heavy-walled and thin-walled. The type of glass to be used is specified below.

The specifications are designed for a maximum operating temperature of 300° C. The length of the sealed tube between the bottom and the start of the taper at the shoulder should be 150 to 175 mm. for the heavy-walled tubes and 180 to 210 mm. for the thin-walled type.

The glass should have a coefficient of linear expansion not exceeding 0.0000040 cm. per cm. per 1° C., with a softening point of 820° C. (Corning Pyrex 7740 or equal). Tubes at one end should have a closed round bottom of about the same wall thickness as the side walls and at the other end should be open and glazed. Tubes must be well annealed. The thickness of the wall and the length depend upon the volume of nitric acid used.

Table I gives the recommended specifications.

Table I. Recommended Specifications

Combustion Tube	Wall Thickness Mm.	O.D. Mm.	Length Mm.	Length of Sealed Tube between Bottom and Start of Taper at Shoulder Mm.	Volume of HNO ₃ (Sp. Gr. at 60° F., Approximately 1.5) Ml.	Temp. $^\circ$ C.
Heavy-walled	2.3 ± 0.3	13 ± 0.8	210 ± 10	150 to 175	More than 0.3 (volume should not exceed 0.7)	250
Thin-walled	1.2 ± 0.2	13 ± 0.7	240 ± 10	180 to 210	0.3 or less	300

16-mm. inside diameter and 225 mm. long. The wells should be held at a fixed inclined position of approximately 45° or should be adjustable. A device for pushing the combustion tubes from the individual wells should be provided.

The temperature in the wells must be maintained at approximately 310° C.; the temperature at any point should not vary

- LITERATURE CITED**
- (1) ANAL. CHEM., 21, 651 (1949).
 - (2) Steyermark, Al, *Ibid.*, 22, 1228 (1950).
 - (3) Steyermark, Al, Alber, H. K., Aluise, V. A., Huffman, E. W. D., Kuck, J. A., Moran, J. J., and Willits, C. O., *Ibid.*, 21, 1555 (1949).
 - (4) *Ibid.*, p. 1283.
 - (5) *Ibid.*, 23, 523 (1951).

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